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U.S. PATENT APPLICATION

OF

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FOR

METHODS OF MAKING CARBON FOAMS

## METHODS OF MAKING CARBON FOAMS

### **BACKGROUND OF THE INVENTION**

5 The present invention relates to carbon foams and methods of making carbon foams as well as their use in a variety of products, such as insulation materials and capacitors and fillers for polymers or elastomers.

Carbon aerogels have been used in a wide variety of applications, including thermal insulation, conducting fillers for polymers or plastics, electrodes, and electrochemical cells. Carbon aerogels which have high surface areas and high DBP values are desirable in many of these and other  
10 applications.

Various methods are known to make carbon aerogels including using pitch, using organic gels with a catalyst and reacting the mixture, using sol-gel polymerization to form highly cross linked networks of high surface area foam and dissolving a polyacrylonitrile in a heated solution with at least one alkali metal halide in a solvent to eventually form a liquid gel. The liquid gel is then heated  
15 wherein the solvent is removed along with the dissolved alkali metal halide to provide a porous form consisting essentially of polyacrylonitrile wherein the porous form is then cured at elevated temperatures to carbonize the polyacrylonitrile to form the carbon foam.

While such methods are useful, there is always a desire in the industry to develop other less expensive methods of making carbon foams which preferably lead to greater flexibility in making  
20 foams having higher surface areas as well as high structure.

### **SUMMARY OF THE PRESENT INVENTION**

A feature of the present invention is to provide carbon foams.

Another feature of the present invention is to provide carbon foams preferably having high  
25 surface areas as well as high structure.

An additional feature of the present invention is to provide inexpensive methods of making carbon foams.

Still, another feature of the present invention is to incorporate the polymer foams into such applications as thermal insulation, conducting fillers, electrodes, reinforcing fillers, and the like.

5 Additional features and advantages of the present invention will be set forth in part in the description which follows, and in part will be apparent from the description, or may be learned by practice of the present invention. The objectives and other advantages of the present invention will be realized and obtained by means of the elements and combinations particularly pointed out in the written description and appended claims.

10 To achieve these and other advantages, and in accordance with the purposes of the present invention, as embodied and broadly described herein, the present invention relates to a method of making carbon foam which involves rapid pyrolyzing at least one pyrolyzable material in the presence of a sufficient amount of at least one oxidizing source to obtain the carbon foam. The pyrolyzable material may comprise the fuel source or a separate source may be used.

15 The present invention further relates to carbon foam made by the above-described method.

The present invention further relates to carbon foam having cells bordered by thin sheets or windows and/or struts. Typically the cells are not closed, but have openings between them. Because the foam can be rigid, pieces of the foam can be broken off, and material can consist of both the foam particles and their fragments.

20 It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are intended to provide a further explanation of the present invention, as claimed.

#### **DETAILED DESCRIPTION OF THE PRESENT INVENTION**

25 The present invention relates to carbon foams and methods of making these carbon foams. The present invention further relates to the use of these carbon foams in a variety of end-use

applications.

The carbon foam of the present invention is preferably made by pyrolyzing at least one pyrolyzable material in the presence of a sufficient amount of at least one fuel source and at least one oxidizing source. The pyrolyzable material or the products of the pyrolysis may provide the fuel source  
5 and/or a separate fuel source may be used. This carbon foam can then be used in its foam state or can be reduced into pieces to form particles which preferably are highly structured.

In more detail, and with reference to the above-described method, the pyrolyzing material or substance is preferably an organic substance such as an organic compound. Examples include, but are not limited to, coal, hydrocarbons, and carbohydrates. Essentially, the substance used is a substance  
10 which will pyrolyze instead of evaporate when heated. A preferred pyrolyzable substance is sugar, cellulose compounds, coal, and the like. Other examples include, but are not limited to, hydrocarbons and polymers and derivatives thereof.

The fuel source that is used in the present invention can be any fuel source, such as a gas, liquid, or solid or combinations thereof. As stated, the pyrolyzable material can serve as the fuel source  
15 and/or at least one fuel source other than the pyrolyzable material can be used. Examples of suitable fuel sources include, but are not limited to, natural gas or hydrocarbon (e.g., oil), or mixtures of two or more materials. An example of such a mixture is a suspension of ground coal in oil.

The oxidizing source can also be in any state, such as a gas, solid, or a liquid, or combinations thereof. Preferably, the oxidizing component is in the form of a gas and is preferably air or oxygen or  
20 both.

The amount of each component in the process can be varied depending upon the desired structure of the carbon foam as well as the desired surface area of the carbon foam. For purposes of the present invention, the heat generated during the method should be sufficient to at least partially pyrolyze the pyrolyzable material. The oxidizing source should be sufficient to at least partially  
25 combust the fuel but the amount of oxidizing source should be controlled such that the pyrolyzable material does not completely combust or burn the pyrolyzable material. The amount of oxidizing

material needed is in the range of 0.05 to 0.75 of the theoretical amount needed to completely burn all of the fuel and combustible material. In the case where a separate fuel is used, the amount of fuel is such that the fuel combustion consumes between 0 and 100 % of the oxidizing material.

The preferred rapid pyrolyzing can occur in any device conventionally used for combustion  
5 such as a carbon black reactor or other suitable combustion chamber.

Examples of pyrolyzing temperature ranges include, but are not limited to, from about 200° C to about 1600° C, and more preferably from about 400° C to about 1600° C. Certainly, other temperature ranges are possible depending upon the type of pyrolyzable substance used. Once the pyrolyzable substance is determined, determining the necessary pyrolyzing temperature is within one  
10 skilled in the art in view of the present application and routine experimentation.

In following the method of the present invention, a variety of orders can be followed with respect to the introduction of the various components. For instance, the fuel source can be used as a carrier stream to introduce the pyrolyzable material into the combustion chamber. Likewise, the oxidizing source can be used as a carrier stream to introduce the pyrolyzable material. For instance,  
15 when the fuel source is a gas stream such as natural gas, a suspension of finely ground particles of a pyrolyzable material, such as coal, can be introduced by this gas stream into the combustion chamber for purposes of the pyrolyzing step. Examples of alternative embodiments include the introduction of the pyrolyzable material into a combustion chamber wherein the fuel source and/or the oxidizing source are already present in the combustion chamber. In this embodiment, the fuel source and the  
20 oxidizing can be pre-ignited prior to introduction of the pyrolyzable material which can be introduced by itself or by a carrier gas or liquid which can be the same or different from the fuel source and/or oxidizing source or can be a neutral source such as nitrogen gas and the like. If the fuel source and oxidizing source are pre-ignited, the pyrolyzable material can be, for instance, injected into the flame for purposes of the pyrolyzing step. Mixtures of solid and liquid materials such as coal in oil can be  
25 used to facilitate the injection. As another embodiment, the fuel source, oxidizing source, and the pyrolyzable material can be mixed all together prior to introduction into the combustion chamber, or

they can be introduced sequentially, or in any other combination or order as along as the pyrolizable material is at least partially pyrolized to form carbon foam.

In other embodiments of the present invention, additional fuel sources and/or oxidizing sources and/or additional pyrolizable material, which can be the same or different from the components already present, can be added. For instance, a fuel source and an oxidizing source can be present in the chamber and the pyrolizable material can be introduced by means of a carrier stream which can also be a second fuel source and/or an oxidizing source. In addition, a fuel source and an oxidizing source can be introduced into a chamber along with pyrolizable material and then additional amounts of a fuel source or an oxidizing source or pyrolizable material, which can be the same or different from the original fuel source, oxidizing source, and/or pyrolizable material can be added. Thus, various modifications can be made to the method of the present invention to meet the desired parameters of the carbon foam.

Any means of introducing a liquid or gas stream can be used for purposes of the present invention in introducing the fuel source and the oxidizing source. Similarly, any means that are conventional with respect to the introduction of particles into a combustion chamber such as by a carrier gas or the like can be used for purposes of the present invention.

After the pyrolizing step, the carbon foam that is formed can be recovered by any conventional technique used to recover particles from a pyrolization including, but not limited to, cyclone separators, fabric filters, and the like.

The carbon foam of the present invention can be subsequently chemically modified or surface treated using a variety of techniques such as the attachment of organic groups onto the surface of the carbon foam. Such techniques and groups include, but are not limited to, these described U.S. Patent Nos. 5,900,029; 5,895,522; 5,885,335; 5,851,280; 5,837,045; 5,803,959; 5,672,198; 5,571,311; 5,630,868; 5,707,432; 5,803,959; 5,554,739; 5,698,016; 5,713,988; WO 96/18688; WO 97/47697; WO 97/47699, and U.S. Patent Application Nos. 09/317,287; 60/116,500; 60/135,558; 60/163,716; and 60/163,857; where all are incorporated herein in their entireties by reference.

The carbon foam, once formed, can be used as any other conventional carbon foam. For instance, the carbon foam can be used in a manner as described in U.S. Patent Nos. 5,300,272; 6,033,506; 5,945,084; and 5,476,878, all incorporated in their entireties by reference herein.

5 The carbon foam can, if desired, be reduced to fragments and/or particles. The reduction of the carbon foam can be achieved by any technique such as grinding the carbon foam into highly structured particles and/or fragments. The grounded carbon foam can be used as filler or any other use for carbon based particles.

10 As indicated above, the carbon foams can be used in a wide variety of application, including thermal insulation, reinforcement applications, fillers for elastomers or polymers such as plastics, electrodes in electrochemical cells or supercapacitors, and the like. The manner and amount of the carbon foam used in each of these applications would be conventional and known to those skilled in the art in view of the present application and the conventional technology of using carbon foam in these applications.

15 The carbon foam formed can be used in various end use applications, such as in polymer compounds, elastomer compounds, thermal insulating materials, electrodes, capacitors, fuel cells, batteries, and the like. The carbon foam can be used in lieu of conventional fillers, pigments, or particles that are used in such end use applications.

20 Other embodiments of the present invention will be apparent to those skilled in the art from consideration of the present specification and practice of the present invention disclosed herein. It is intended that the present specification and examples be considered as exemplary only with a true scope and spirit of the invention being indicated by the following claims and equivalents thereof.